

REMOVAL OF IBUPROFEN FROM WATER USING MAGNETIC Fe₃O₄-Ag₂O/CELLULOSE AS AN ADSORBENT

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ABSTRACT

Ibuprofen is widely used to reduce minor aches and fevers from a various condition such as the common flu. The increase in the waste of ibuprofen may cause contamination and raise a serious problem in the water effluence. It is needed to reduce the waste of ibuprofen by the adsorption process. Both cellulose from empty fruit bunch and modified bimetal oxides Fe₃O₄-Ag₂O were synthesized and characterized as magnetic Fe₃O₄-Ag₂O/cellulose adsorbent. The BET surface area was 177.7 m²/g with a predominantly mesoporous structure. SEM EDX spectra proved an unsmooth adsorbent with irregular-sized particles. FTIR spectra at 552-693 nm are assigned to Fe-O and Ag-O stretching vibration. XRD pattern analysis shows the crystallized size is 34.16 nm. The highest removal efficiency of ibuprofen was obtained at acidic conditions (pH = 2), a concentration of 200 ppm, and a contact time of 120 min. The equilibrium adsorption data proved that the Freundlich isotherm is more favorable than the Langmuir isotherm model. The kinetic model showed that the pseudo-first-order is a more suitable model than the second-pseudo-order. The result of the experimental data proved that the Fe₃O₄-Ag₂O/cellulose will be a potential adsorbent for ibuprofen removal.

Keywords: Ibuprofen, Fe₃O₄-Ag₂O/cellulose, Adsorbent, Isotherm models, Kinetics models.

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INTRODUCTION

Ibuprofen is a non-steroidal anti-inflammatory, analgesic, and anti-pyretic drug. It works by reducing hormones that cause inflammation and pain in the body such as fever, toothache, headache, etc. Ibuprofen drugs are widely used and easily found in drug stores without requiring a medical prescription. Previous research found that the amount of ibuprofen consumed is approximately 58, 162, and 300 tons per year in the US, UK, and Poland, respectively.^{1,2} A high consumption of ibuprofen has been considered a potential environmental risk.^{3,4} Recently, the presence of pharmaceutical drugs including ibuprofen has been detected, and there is an accumulation of chemical drug waste in the natural environment such as in the aquatic environment. Most drug waste is mainly discharged from dumped in domestic waste, veterinary medical, expired drugs, hospitals, and drug industries.⁵ Ibuprofen drugs can be introduced into the ecosystem through different ways released on the ground in animal urine and feces. The presence of a substantial chemical from drug waste in the ecosystem with higher concentration can cause an environmental hazard and make it highly resistant to biodegradable, toxic persistent food chains.^{6,7} It is essential to implement regulations and monitoring systems to dispose of drug waste to minimize the negative impact on the environment. Some methods have been used for wastewater treatments such as adsorption, photocatalyst, ion exchange, precipitation, and membrane filtration. Adsorption is a common method that is widely used for the separation and purification process. The advantages of the adsorption process are high efficiency, lack of by-products, and effectiveness for liquid waste.^{7,9} The adsorption process usually uses solid materials such as activated carbon, zeolites, biochar, cellulose, and composite. The cellulose molecule consists of high hydroxyl functional groups easily modified with metal oxide. The interaction of the hydroxyl group with metal oxide through ionic exchange may extend the function of the active site as an adsorbent. The development of nanometal oxides loaded onto cellulose as a biopolymer can be considered an advanced nanomaterial in the future due to its renewability and availability in large quantities including excellent physical and chemical properties.^{10,11}

The present work aimed to synthesize and characterize the magnetic Fe₃O₄-Ag₂O/cellulose as an adsorbent from the nanoparticles of bi-metallic oxides Fe₃O₄-Ag₂O immobilized on the surface of natural cellulose.

The application of magnetic Fe₃O₄-Ag₂O/cellulose was investigated for ibuprofen removal from water at various conditions, including the effect of pH, concentration, contact time, and catalyst dose. Analysis of ibuprofen residue was determined using UV-visible spectrophotometry.

EXPERIMENTAL

Material and Methods

Empty fruit bunches (EFB) as solid waste were collected as raw material for cellulose preparation. All chemicals such as ferric chloride hexahydrate (FeCl₃·6H₂O), ferrous chloride tetrahydrate (FeCl₂·4H₂O), sodium hydroxide (NaOH), silver nitrate (AgNO₃), sodium hypochlorite (NaOCl), and Standard Reference for ibuprofen were analytical grade and purchased from Merck.

Preparation of Cellulose

Approximately, 250 gr of dried EFB was cut down into small sizes (1-2 mm) and impregnated into 1 L of 2.5 M NaOH solution at 80°C for 24 h. The mixture was filtered, washed, and neutralized with a hydrochloric acid (HCl). The bleaching process was performed by immersing the fiber into 500 ml NaOCl (12 %) at room temperature for 1 h. The mixture was washed and dried at 50°C for 24 h. For the acid hydrolysis process, the dried fiber was immersed into 100 ml H₂SO₄ (64 %) at room temperature for 1 h, washed with deionized, and then neutralized by NaOH solution. The obtained suspension was separated by centrifuge at 1500-2000 rpm. The solid suspension was sonicated and dried in an oven at 105°C overnight. The cellulose obtained was kept for further analysis.

Preparation of Fe₃O₄-Ag₂O/Cellulose

The preparation of Fe₃O₄ particles was carried out by the mixture of FeCl₃·6H₂O (27.033 g) and FeCl₂·4H₂O (9.942 g) in 100 ml of deionized water. The mixture was slowly heated to 60°C and added strong base NaOH to pH 10-11. The precipitation as a Fe₃O₄ particle was collected by washing it with deionized water and ethanol and then dried at 105°C for 24 h in an oven. The magnetic Fe₃O₄ was collected using a magnetic bar. The preparation of Ag₂O particles was carried out by the dissolving of AgNO₃ (5.001 g) in 100 ml deionized water. The solution was heated to 60°C and added with a strong base NaOH solution to pH 9-10. The precipitation as Ag₂O particles were collected and washed with deionized water and ethanol and then dried in an oven at 105°C for 24 h. The Fe₃O₄-Ag₂O/cellulose was prepared by a mixture of 15 g of cellulose, 10 g of Fe₃O₄, and 5 g of Ag₂O in the hydrothermal reactor. The mixture was immersed in 50 ml of deionized water and 25 ml of ethanol in the hydrothermal reactor. A nitrogen gas was added into the reactor, tightly closed, and slowly heated to 350°C for 3 h. After cooling down to room temperature, the mixture was washed with deionized water, and dried at 110°C overnight. The result is magnetic Fe₃O₄-Ag₂O/cellulose and kept for further characterization.

Adsorption Study for Removal of Ibuprofen

The rate of ibuprofen removal was determined at room temperature under UV light (100 watts). Fe₃O₄-Ag₂O/cellulose was applied as an adsorbent to remove ibuprofen in water by the photocatalytic process using UV light 100 watts. Before the experiment, a stock solution of ibuprofen was prepared by dissolving 1.0 g ibuprofen into 1000 ml ultrapure water in a volumetric flask and calibrated at different concentrations of 2-12 ppm. Adsorption studies were performed at different pH of ibuprofen (pH = 2-8), concentrations (100-400 ppm), and contact times (30-120 min). The residue of ibuprofen was determined at the maximum absorbance of 224 nm using the UV-visible spectrophotometer. The percentage removal of ibuprofen was calculated by the following eqns.-1 and 2.

$$\text{Removal (\%)} = \frac{C_0 - C_t}{C_e} \times 100\% \quad (1)$$

$$qt = \frac{(C_0 - C_t) V}{W} \quad (2)$$

Where Co and Ct are the initial concentration (mg/L) and concentration (mg/L) at t (min), respectively. qt is adsorption capacity. The V and W are the volume (L) and weight of adsorbent (g), respectively.

RESULTS AND DISCUSSION

Characterization

The nitrogen adsorption-desorption isotherm at 77 K and the relative pressure at 0.05-0.99 were used for determining the BET surface area shown in Fig.-1. The isotherm model exhibits a wide hysteresis loop and isotherm type IV corresponding to the mesopores. BET surface area, total pore volume, and the average pore size of the composite are 177.7 m²/g, 0.08 cc/g, and 8.03 nm, respectively.

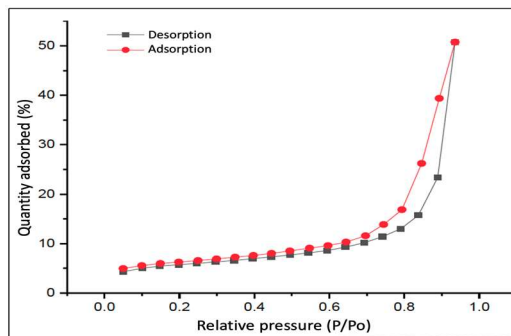


Fig.-1: The Nitrogen Adsorption-desorption Isotherm (77 K) of Fe₃O₄-Ag₂O/Cellulose

FTIR spectra of cellulose, Fe₃O₄-Ag₂O, and Fe₃O₄-Ag₂O/cellulose are shown in Fig.-2. The cellulose functional groups were obtained at wave number 3336-3447 nm related to the O-H stretching vibration. A weak peak at 2907 nm is related to the C-H stretching vibration from the cellulose functional group. Strong peaks at around 1633-1639 nm are assigned to the C=O stretching vibration of an aromatic ring. The weak bands at around 1417 and 1316 nm are assigned to the C=C and C-O stretching vibrations. The bands at 1187-990 nm are attributed to the C-O stretching vibration. The FTIR spectrum of Fe₃O₄-Ag₂O appeared at 552-693 nm which can be assigned to Fe-O and Ag-O stretching vibration.¹² These results proved that metal ions Fe and Ag were strongly bonded to oxygen through chemical interaction.

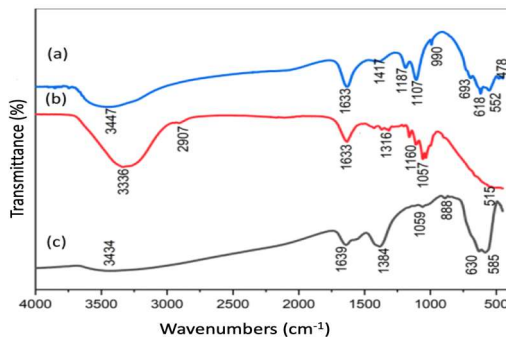
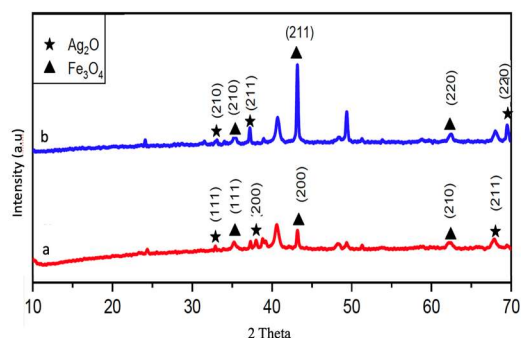


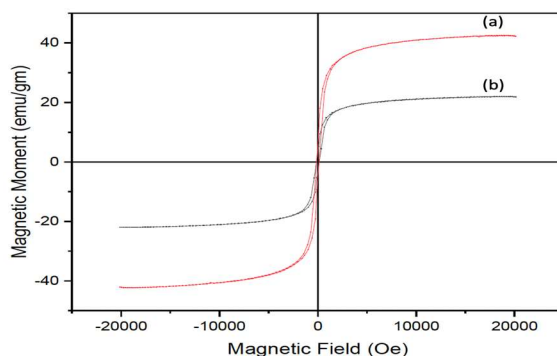
Fig.-2: FTIR Spectrum of (a) Fe₃O₄-Ag₂O/Cellulose, (b) Cellulose, and (c) Fe₃O₄-Ag₂O

Comparison of XRD pattern of cellulose, Fe₃O₄-Ag₂O, and Fe₃O₄-Ag₂O/cellulose in the range of 2θ 10°-70° are shown in Fig.-3. The XRD pattern of Fe₃O₄-Ag₂O is shown in 2θ = 30.3°, 35.6°, 43.8°, 57.3°, and 63.8° (JCPDS 00-019-0629) relatively corresponding to (111), (111), (002), (210), and (102) for Fe₃O₄; and 2θ = 32.9° and 38.2° for Ag₂O (JCPDS 01-0751532).^{13,14} The crystalline phase is a simple cubic with an average crystallized size of 34.16 nm. XRD pattern of Fe₃O₄-Ag₂O/cellulose is shown by one peak at 2θ = 24.3° related to the amorphous cellulose.

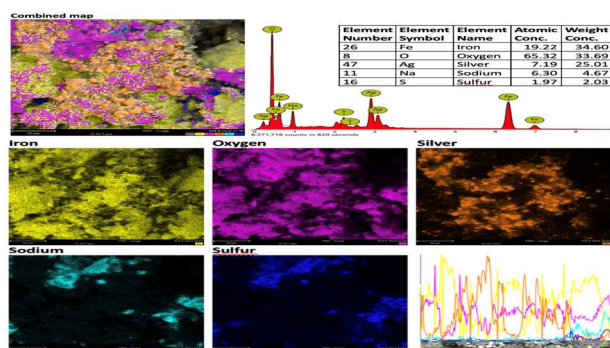
The comparison of magnetic properties between Fe₃O₄-Ag₂O and Fe₃O₄-Ag₂O/cellulose was determined by the vibration sample magnetometer magnetic (VSM) and displayed in Fig.-4. Magnetic properties were evaluated from the hysteresis curve which was differentiated into the magnetic saturation (Ms), field coercivity (Hc), and the remanent magnetization (Mr). The saturation magnetization indicates the ability of nanoparticles to maintain irreversibility magnetic domains from the outside of the magnet. The coercivity field of the magnetization is zero.^{15,16} The magnetic remanent is the ability when it is given an external field. The result shows that the magnetic saturation (Ms) of Fe₃O₄-Ag₂O is 42.21 emu/g which decreased to 21.93 emu/g after modification with cellulose (Fe₃O₄-Ag₂O/cellulose).

Fig.-3: XRD Pattern of (a) Fe₃O₄-Ag₂O and (b) Fe₃O₄-Ag₂O/Cellulose

The lower Ms for Fe₃O₄-Ag₂O/cellulose is due to the surface of Fe₃O₄ being closed by the cellulose matrices. The remanent magnetization of the Fe₃O₄-Ag₂O (17.85 emu/g) is higher than Fe₃O₄-Ag₂O/cellulose (6.50 emu/g) due to the size effect of the particle. The value of coercivity is zero. These results proved that Fe₃O₄-Ag₂O/cellulose has a strong magnetization, small coercivity, and low magnetic remanence that can be used as an adsorbent for wastewater treatment.

Fig.-4: Magnetization Curve of (a) Fe₃O₄ and (b) Fe₃O₄-Ag₂O/Cellulose

SEM microstructure of Fe₃O₄-Ag₂O/cellulose was displayed in Fig.-5. The external structure of the composite shows an irregular surface with low porosity. The EDX analysis shows the distribution of atoms on the surface composite. The elemental analysis result is arranged based on the different colors consisting of the main elements loaded on the surface.¹⁷ This study reported that oxygen, iron, and silver are the main elements distrusted on the surface of cellulose. However, other atoms such as sodium and sulfur are predicted as impurities of atoms attached to the surface. The result shows that the ionic iron and silver are strongly bonded with cellulose with high oxygen contents.

Fig.-5: Distribution of Atoms of Fe₃O₄-Ag₂O/Cellulose

Removal of Ibuprofen Study

The percentage of ibuprofen removal obtained at different pH levels is displayed in Fig.-6(a). The general trend shows that the maximum curve was obtained at acidic conditions (pH 2) with a percentage removal of 75.53 % and then decreased to alkaline conditions (pH 8) with the percentage removal of 61.58 %. The

reason for this adsorption process can be described that in acidic conditions, the highest interaction occurred through an electrostatic positive charge of $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ and a negative charge of ibuprofen. Conversely, in alkaline conditions, adsorption efficiency decreased due to the repulsion of the interaction between $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ and ibuprofen as a reduction of positive and negative charges.

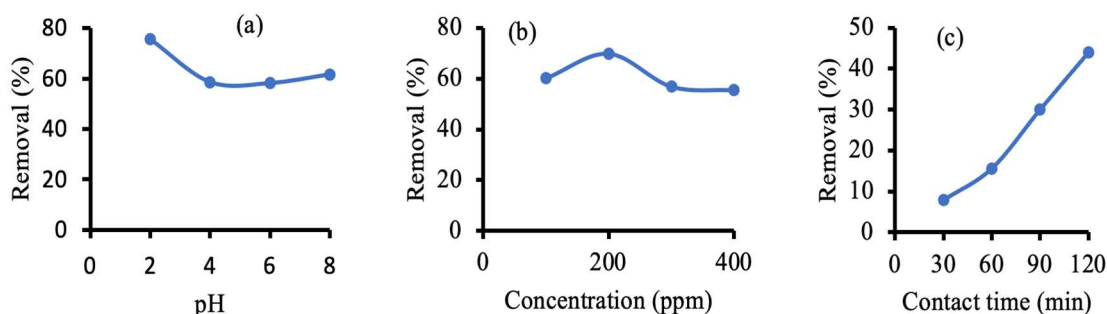


Fig.-6: Percentage Removal of Ibuprofen at Different Conditions, (a) pH, (b) Concentrations, and (c) Contact Times

The effect of a concentration on the adsorption using $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ is shown in Fig.-6(b). The optimum curve removal was obtained at 200 ppm (65.77%) and decreased at the increase in concentrations of ibuprofen toward 400 ppm (55.43%). This reason is that at initial concentration (200 ppm), the negative charge of ibuprofen was strongly attracted to the active site of adsorbent toward higher concentration (400 ppm). However, at a higher concentration (600-800 ppm), both compounds' active sites have reduced due to the active sites' interaction. Therefore, the percentage of ibuprofen removal decreased to 55.43 %. The effect of contact times on the ibuprofen removal is displayed in Fig.-6(c). The trend of the adsorption process significantly increased with an increase in the contact times starting from 10 min (7.90 %) to 120 min (44.01 %). This result shows that the longer contact times in the adsorption process, the more interaction between active sites of both compounds resulting in a strong attraction toward a higher percentage removal of ibuprofen.

Adsorption Isotherm

Analysis of the adsorption isotherm of ibuprofen using $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ was applied by two models: Langmuir and Freundlich isotherms. The constant of the Langmuir equation was determined by linear slope using eqn.-3.

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (3)$$

Where C_e and K_L are the equilibrium concentration (ppm) and the Langmuir constant related to the free adsorption energy, respectively. q_e and q_m are the adsorption capacity (mg/g) and the monolayer adsorption capacity (mg/g), respectively. The plot of $1/q_e$ versus $1/C_e$ should be linear. The values of K_L and q_m can be measured from the slope and intercept of the plot, respectively.

The Freundlich model was used through the linearized slope as following eqn.-4.

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

Where, K_F is a constant of Freundlich isotherm of the adsorption capacity (mg/g) and the constant $1/n$ relates to the intensity of adsorption. The plot of $\log q_e$ versus $\log C_e$ should be linear and the value of K_F can be measured from the intercept and slope of the line, respectively.

The curve of the Langmuir and Freundlich isotherm models is shown in Fig.-7. The isotherm models can be determined from the value of the correlation coefficient (R^2). The parameters of isotherm and Freundlich values are displayed in Table-1. It was observed that the R^2 value for the Freundlich is closer to 1 than the value of R^2 for Langmuir. It can be concluded that the removal of ibuprofen corresponds to the Freundlich isotherm model which is popular with the physisorption model through multilayer adsorption. This result can be confirmed with the value of n related to the adsorption process occurring between ibuprofen and $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$. The higher the value of n ($n > 1$), the weaker the interaction between adsorbate and

adsorbent (multilayer), conversely if the value of n is smaller than 1 ($n < 1$), the higher the interaction between ibuprofen and $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ (monolayer). The value of constant (K) indicates the level of affinity occurring between ibuprofen and $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$. If the value of K_L is higher than 1 then the level of affinity of $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ is strong enough.¹⁸ However, if the value of K_L is less than 1 indicates the level of $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ has a weak affinity. Based on this data, the values of $n < 1$ and $K_L < 1$ are more favorable multilayer.

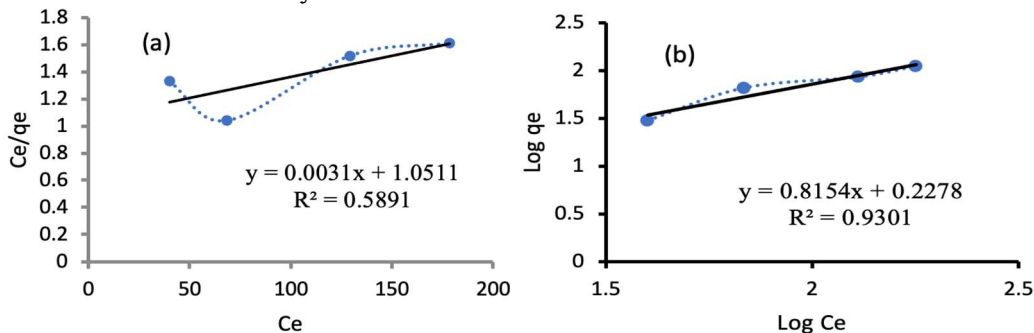


Fig.-7: The Curves of Langmuir (a) and Freundlich (b) Isotherm for Removal of Ibuprofen using $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/Cellulose}$

Table-1: Parameter of Langmuir and Freundlich Isotherm Models

Adsorbate	Langmuir isotherm			Freundlich isotherm		
	q_m (mg/g)	K_L (L/mg)	R^2	K_F	n	R^2
Ibuprofen	263.157	0.9466	0.5891	2.294	1.322	0.9301

Adsorption Kinetic Study

The mechanism of the adsorption process between ibuprofen and $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ adsorbent was evaluated from two approaches to the adsorption kinetic models: pseudo-first-order and pseudo-second-order.¹⁹ The pseudo-first-order commonly called Lagergren kinetic is widely used for studying the liquid-solid adsorption system.^{20,21} The pseudo-first-order model can be expressed as the following eqn.-5.

$$\log (q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (5)$$

Where, q_e and q_t are the amounts of concentration adsorbed (mg/g) at the equilibrium and at any time, t (min), respectively. The k_1 (1 min^{-1}) is the rate constant of the adsorption obtained from the plot of $\log (q_e - q_t)$ versus t .

The pseudo-second-order models can be expressed as the following eqn.-6.

$$\frac{1}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (6)$$

Where, k_2 is the Freundlich constant determined from the linear plot of t versus t/q_t and related to the value of q_t and the intercept. The plot of the kinetic model on the adsorption process of ibuprofen and $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ is shown in Fig.-8.

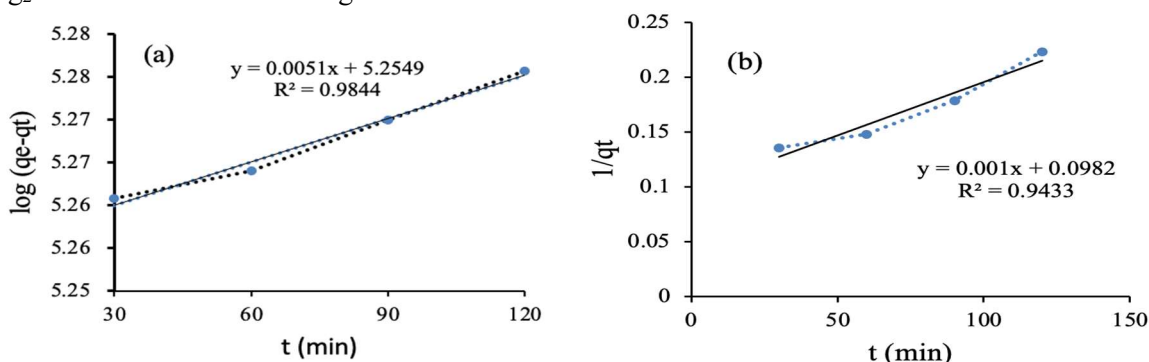


Fig.-8: Kinetic Models (a) Pseudo-First-Order and (b) Pseudo-Second-Order Models

The parameter of the kinetic study is shown in Table-2. The pseudo-first-order is a more favorable model with R^2 of 0.9844 related to the previous research concerning the presence of Fe_3O_4 on the cellulose. In this case, the pseudo-first-order has limited adsorption with physisorption adsorption. The pseudo-second-order has a lower value of $R^2 = 0.9433$ corresponding to the chemisorption.²²

Table-2: Parameter of Pseudo-First-Order and Pseudo-Second-Order

Adsorbate	Pseudo-first-order			Pseudo-second-order		
	q_m (mg/g)	K_L (L/mg)	R^2	K_F	q_e (mg/g)	R^2
Ibuprofen	191.5023	0.0051	0.9844	1.03×10^{-5}	1000	0.9433

CONCLUSION

In this study, the magnetic adsorbent of $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ was investigated. The characterization of $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ was evaluated. The application of ibuprofen removal was carried out at different adsorption parameters including the effect of pH, concentrations, and contact times. The maximum percentage of ibuprofen removal was obtained at the acidic conditions at pH 2, a concentration at 200 ppm, and a contact time of 120 min. The equilibrium adsorption data was evaluated from the best value of a correlation coefficient (R^2). The Freundlich isotherm model is a more favorable model with a better value of R^2 compared to the value of R^2 of the Langmuir isotherm. The kinetic model of adsorbate-adsorbent interaction shows a best-fit line by the pseudo-first-order ($R^2 = 0.9844$) compared to the pseudo-second-order ($R^2 = 0.9433$). Based on the presented data, the $\text{Fe}_3\text{O}_4\text{-Ag}_2\text{O/cellulose}$ adsorbent can be regarded as a potential adsorbent for the removal of ibuprofen in an aqueous solution which can be used to control water pollution.

CONFLICT OF INTERESTS

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID IDs, given below:

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